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High Performance Fibers for Structurally Reliable Metal and Ceramic Composites

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STRUCTURALLY RELIABLE METAL AND CERAMIC
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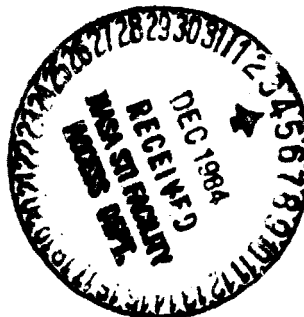
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HIGH PERFORMANCE FIBERS FOR STRUCTURALLY RELIABLE METAL AND CERAMIC COMPOSITES

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INTRODUCTION

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Motivated by aerospace applications requiring composite materials that are lightweight, stiff, and strong, fiber research and development efforts over the last two decades have resulted in a variety of commercially available "high performance" continuous fibers with low densities (<4 gm/cc), high Young's moduli (>200 GPa), and high tensile strengths (>2 GPa). However, for certain applications requiring high structural reliability, very few of these fibers are ideally suited as reinforcement for either metal matrix composites (MMC) or ceramic matrix composites (CMC). That is, in their current production state, these commercial fibers fail to meet all the necessary property requirements for providing MMC or CMC with high strength and toughness. It is the purpose of this paper to discuss these fiber property requirements in general and then to present two examples of how they are influencing commercial fiber evaluation and improvement studies at NASA which are aimed ultimately at developing structurally reliable MMC and CMC for advanced gas turbine engines.

FIBER PROPERTY REQUIREMENTS

Strong and Tough MMC

Assuming a ductile metal matrix, fiber-controlled composite fracture, and unidirectional composite loading, the general microstructural requirements for strong MMC are the alignment in the load direction of a high volume fraction of high strength fibers which are equally strained under composite loading. This implies that the reinforcing fibers must be continuous (high length to diameter ratio), be strongly bonded to the matrix (for good load transfer), possess a high as-produced tensile strength, and be capable of retaining a large percentage of this strength under the fabrication and service conditions typically encountered with the MMC.

Strength retention problems of direct concern are those related to fiber abrasion during composite processing and to fiber-matrix reaction during high temperature composite processing and use. Because a high performance fiber typically fractures in a brittle manner, the reaction problem can degrade composite strength not only by creating new strength controlling flaws on the fiber surface but also by forming these flaws with a high density along the fiber length. High flaw density implies a greater probability that neighboring fibers will have critical flaws on the same transverse plane so that upon individual fiber fracture, the composite is more likely to experience a low-strength cleavage-type of fracture.

Another strength degradation problem of major concern is that related to fiber-matrix thermal expansion mismatch which on composite thermal cycling can introduce undesirable residual stresses and subsequent in situ fiber fracture

or fiber-matrix debonding. Thus, if possible, the reinforcing fiber should have an expansion coefficient close to that of the matrix.

For tough MMC, the general microstructural requirements are that high stresses be needed to fracture fibers at the tip of composite cracks and that the stress concentrations at the crack tips be as low as possible. The first requirement again implies the need for fibers with high in situ tensile strength with a low linear density of critical flaws. The second requirement implies that full advantage should be taken of the crack blunting ability of the ductile metal matrix. This can be achieved by maximizing the interfiber spacing without reducing fiber volume fraction. Since interfiber spacing increases with increasing fiber diameter, it follows that tough MMC should contain very strong fibers with as large a diameter as possible within the dimensional constraints of the particular application.

High in situ strength and large diameter are also important in achieving MMC with high energy absorption capability under mechanical impact. The high fiber strength allows the fibers to absorb maximum elastic energy before fracturing. The large fiber diameter allows the ductile matrix between fibers to contribute maximum plastic energy absorption under the shear-type deformation modes often encountered under impact conditions (refs. 1 and 2).

Strong and Tough CMC

Typical CMC macroscopic fracture is not initiated by fiber fracture as in MMC but by the low-strain propagation of inherent microcracks within the as-produced brittle matrix. This difference in fracture mechanism results in significantly different fiber properties required for achieving high strength and high toughness CMC.

To illustrate this, consider figure 1 which is a schematic of the stress-strain curves typically encountered for monolithic ceramics and for CMC with continuous reinforcing fibers aligned along the load direction. For an unreinforced monolithic matrix with elastic modulus E_m , low temperature fracture is initiated by processing-related bulk flaws at an average applied strain ϵ_m and an average applied stress $\sigma_m = E_m \epsilon_m$. For the composite, if the fibers have a higher modulus than E_m , the CMC modulus will be higher and the CMC stress required for matrix flaw propagation will also be higher provided the CMC fracture strain ϵ_c is equal to or greater than the monolithic fracture strain ϵ_m .

One method of achieving high composite fracture strain is to avoid the formation of matrix flaws during composite processing that are appreciably larger in size than the flaws typically formed during monolithic matrix processing. Another method is to have a high concentration of fibers bridge the matrix microcracks (cf. insert, fig. 1), so that under composite loading, the bridging fibers will reduce crack openings, thereby requiring greater applied strains for matrix crack propagation than those needed for the same cracks in the unreinforced matrix (ref. 3).

Once matrix crack propagation is initiated, if the fiber-matrix interfacial bonding is strong, the stress concentration on fibers at the crack tip will generally be high enough to fracture the fibers, thereby resulting in a low-toughness cleavage-type of CMC fracture. However, if the interfacial

bonding is weak, matrix cracks will propagate around the fibers and not through them. If the load carrying ability of the fibers is high enough to support the full applied load, then as each critical matrix crack propagates to the composite surface, the composite will elongate but will not fracture catastrophically. The resulting CMC fracture will then appear as a series of macroscopic matrix cracks that are bridged by the reinforcing fibers (ref. 4) (cf. insert, fig. 1).

In light of the above description of CMC deformation and fracture, it follows that strong CMC should contain a high volume fraction of fibers that are continuous, are stiffer than the matrix, and possess a small diameter. The first two properties serve to increase composite stiffness, thereby increasing the stress level for macroscopic composite fracture. Under elastic deformation conditions at low temperature, high fiber stiffness implies high modulus; whereas under time-dependent deformation conditions at high temperature, high fiber stiffness implies both high modulus and high creep resistance.

The high volume fraction and small diameter properties are needed to insure that a sufficient number of fibers bridge the processing-related matrix microcracks, thereby preventing their propagation until higher applied strains. An additional performance advantage of a small diameter fiber is that the fiber length required for full fiber-matrix load transfer (critical transfer length) is also small. As interfacial bonding in CMC is reduced from strong to weak, this critical length increases so that the smaller the fiber diameter, the more likely that maximum composite performance (e.g., stiffness) will be achieved even in CMC of small dimensions.

Another argument for small diameter fibers in CMC is that the risk of introducing processing related flaws that are larger than the typical inherent matrix flaws should be reduced (ref. 5). These new flaws could arise from the low strain fracture of individual fibers weakened during composite processing or perhaps from unconsolidated matrix material between fibers. In either case, the size of the new flaws might be expected to be close to that of the fiber diameter.

For tough CMC, it is important that the high strain mechanism in which fibers bridge macroscopic matrix cracks be allowed to operate. This implies that the CMC should contain a high volume fraction of fibers that are weakly bonded to the matrix, are continuous, possess a high in situ tensile strength, and are capable of retaining a high percentage of this strength under oxygen and other service environments to which the CMC will be exposed.

Summarizing the above fiber property requirements (cf. fig. 2), high performance fibers for structurally reliable MMC and CMC should in general be continuous, be capable of high volume alignment in the load direction, possess a high as-produced tensile strength, be capable of retaining this strength under composite fabrication conditions, and possess a thermal expansion close to that of the matrix. For MMC, the fibers should also be strongly bonded to the matrix and have as large a diameter as possible. On the other hand, the fibers for CMC should possess a high modulus, good creep resistance, a small diameter, weak matrix bonding, and high strength retention in oxygen and other CMC service environments. Clearly the achievement of some of these fiber properties is dependent on the chemical and physical character of the matrix. However, the majority are generally matrix independent (indicated by asterisks in fig. 2).

FIBER EVALUATION AND IMPROVEMENT STUDIES

Fiber research efforts at NASA Lewis Research Center have been aimed at developing structurally reliable MMC and CMC for application in advanced gas turbine engines. The general approach has been first to evaluate promising commercially available fibers against the property guidelines discussed above and then, if possible, improve the fibers in those areas of property deficiency. Two areas of recent interest have been (1) the strength improvement of large diameter boron fibers for tough and impact resistant boron/aluminum composites and (2) the evaluation of silicon carbide (SiC) fibers for strong and tough ceramic matrix composites with service temperatures as high as 1400 °C.

Strength Improvement of Large Diameter Boron Fibers

Due to their excellent stiffness-to-weight and strength-to-weight ratios, B/Al composites are being considered as a structural replacement for titanium in the first stage fan blades of advanced gas turbine engines. However, with the commercial standard 142 μm diameter boron fiber, B/Al toughness and impact energy absorption values are generally less than desirable for this toughness-critical application. From the above fiber property discussion, one approach to improving MMC toughness is to develop stronger and larger diameter fibers than the commercial standard, thereby allowing both the fibers and matrix to absorb a greater amount of strain energy before and during composite fracture. Because boron fibers are produced by chemical vapor deposition (CVD), larger diameter fibers have been made commercially but have generally displayed tensile strengths equal to or less than 3.8 GPa, the value typically displayed by the standard 142 μm diameter fiber. Thus the problem existed as to whether processing methods could be developed for the large diameter fibers which would significantly improve their strengths.

To solve this problem, basic deformation and fracture studies (ref. 6) were performed on some limited-production 200, 280, and 400 μm diameter boron fibers with as-produced average tensile strengths ranging from 2.1 to 4.0 GPa and relatively high coefficients of variation (cf. table I). Fracture surface analyses revealed that individual fiber strengths below 4 GPa were controlled by processing-related flaws on the fiber surface and strengths above 4 GPa by the tensile fracture of the 17 μm tungsten-boride core (original tungsten substrate) upon which the boron was deposited by CVD. (This latter type of core-initiated fracture is a good example of the undesirable effects of strong bonding in ceramic/ceramic composites). By combining these fracture observations with the results of auxiliary studies concerning boron physical and chemical behavior, secondary fiber treatments were developed by which the effects of surface and core flaws could be reduced and the strength properties of the large diameter fibers significantly improved.

The simplest treatment for surface flaws was a slight chemical polish which, in smoothing the fiber surface, raised fiber flexural strengths to over 10 GPa and left the core as the only source of fiber tensile fracture (ref. 6). For core-initiated fracture, the average tensile strength for all large diameter fibers was ~4.4 GPa with very low coefficients of variation (cf. table I).

To further strengthen the polished fiber, it was determined that since the core is strongly bonded to the boron sheath, it could be put under internal compression by processing treatments which cause the sheath to experience a permanent axial contraction strain ϵ_z . In this case, the fiber tensile strength increases by an amount $\epsilon_z E_f$ where E_f is the boron fiber modulus. The most cost-effective method for sheath contraction was determined to be an oxidation treatment (ref. 7). Apparently during formation of boron oxide on the fiber surface, boron atoms are removed from within the sheath, thereby causing it to axially contract.

Fiber strength improvement after application of both the oxidation and polishing treatments is shown in figure 3 for two large diameter fibers. It can be seen that average tensile strengths as high as 5.6 GPa can be achieved for 203 and 406 μm diameter fibers. Also, by controlling oxidation-induced contraction below 0.3 percent, fiber tensile strengths can be controlled in the range between 4.4 and 5.6 GPa. Throughout this range, fiber fracture remains core-controlled with a strength COV of less than 5 percent. Above a contraction strain of 0.3 percent, the strength controlling flaw changed from the core itself to low-strength voids created at the core-sheath interface during the oxidation treatment (ref. 7). These voids are apparently produced as boron atoms are removed from the sheath to form the boron oxide coating on the fiber surface.

As indicated in table II, secondary treatments now exist which when applied to commercial 400 μm diameter boron fibers result in values for average tensile strength, strength coefficient of variation, and fracture energy that are significantly better than those displayed by the standard 142 μm fiber. It is anticipated that once this technology is utilized to produce large quantities of fiber, more reliable B/AI composites will be developed with greater strength, higher fracture toughness, and improved impact energy absorption capability.

Evaluation of SiC Fibers for High Temperature CMC

For higher temperature applications such as components in the hot section of advanced heat engines, there has been recent interest in developing CMC that are stronger, tougher, and therefore more reliable than monolithic silicon-based ceramics at service temperatures as high as 1400 °C. These extreme temperatures plus the need for oxidation and creep resistance narrow the commercial fiber choice down to two types of continuous fibers based on the β -SiC microstructure.

One type, the Nicalon fiber from Nippon Carbon Co., is produced by thermal decomposition of a polycarbosilane precursor to form a yarn containing 10 to 20 μm diameter fibers. As indicated in table III, mechanical property results for this fiber taken from the literature (refs. 8 to 10, and 16) show an average modulus of 180 GPa and an average tensile strength of 2 GPa (gauge length \approx 50 mm). The relatively low modulus in comparison with pure polycrystalline β -SiC (420 GPa) is a consequence of the fiber production method which results in a microstructure containing SiO_2 and excess carbon.

These impurities are also responsible for Nicalon's poor strength retention and creep resistance at temperatures above 1200 °C (cf. table III). For example, at these temperatures the SiO_2 and carbon react to evolve CO, thereby

forming strength-limiting microvoids within the fiber (ref. 10). Also the glassy SiO_2 probably contributes to excessive grain boundary sliding. Thus, it would appear at the present time that CMC reinforced by the Nicalon fiber are limited to processing and service temperatures below 1200 °C. However, government and industry-sponsored research efforts directed toward improved polymer-derived SiC fibers are currently in progress. These are aimed both at developing new fiber types with reduced impurity content and also at determining possible matrix and/or CMC processing conditions which minimize or delay until higher temperatures CO evolution in the Nicalon fiber.

A second type of commercial β -SiC fiber is that produced by Avco Specialty Materials Inc. utilizing chemical vapor deposition of silicon and carbon-containing compounds onto a 37 μm graphite-coated carbon substrate (cf. fig. 4). The 142 μm diameter of this fiber type is measurably greater than that of the Nicalon fiber. However, because the CVD production method results in a relatively pure microstructure, the Avco fibers display a significantly greater modulus (400 GPa) and as-produced strength (~4 GPa) (refs. 11 and 12). In addition, because of the CVD production method the manufacturer is able to deposit various types of silicon and carbon-containing coatings on the base SiC fiber which could be beneficial in retarding environmentally-induced fiber degradation at high temperatures.

To evaluate this coating factor as well as the intrinsic strength stability and creep resistance of the CVD SiC fibers, commercial Avco fibers with two types of coating were subjected to high temperature deformation and fracture studies in a variety of gaseous environments (refs. 12 and 13). The silicon content for the two carbon-rich coatings are shown schematically in the lower portion of figure 4. The standard Avco SiC fiber has a coating in which the silicon/carbon ratio increases from zero at the fiber surface to unity within a thickness of ~1 μm (ref. 14). The coating for the Avco SCS-6 fiber is thicker (~2 μm) and also contains a higher Si content at the fiber surface due to the formation of SiC microcrystals (refs. 14,15).

The tensile strength retention results for the Avco fibers heat treated for 15 min in argon or nitrogen (ref. 12) are shown in figure 5. The results for strength at temperature in an oxygen environment (ref. 12) are shown in figure 6. For both data sets, fiber test length was 50 mm. For comparison purposes, each figure includes the approximate strength behavior for the Nicalon fiber. These results show clearly that the CVD fibers perform significantly better than Nicalon. It is also evident that the SCS-6 fiber with its SiC surface coating has better strength retention than the standard SiC fiber. This could be attributed to better resistance to oxygen attack or possibly to reduced thermally-induced microstructural changes (ref. 12).

Regarding the projected use of these fibers for CMC, if strength degradation does not occur by matrix contact, the figure 5 results suggest that for short times in inert atmospheres at CMC processing temperatures as high as 1600 °C, the SCS-6 fiber should retain low temperature tensile strengths over 1.2 GPa and fracture strains over 0.3 percent. This implies that at the onset of matrix crack propagation, which at low temperature typically occurs below 0.2 percent strain, the SCS-6 fibers should remain intact and therefore capable of macrocrack bridging (cf. fig. 1). In addition, if the CMC should macrocrack in an oxygen environment, the figure 6 results suggest that the SCS-6 fiber should retain a short time tensile strength greater than 1 GPa for a 1400 °C service temperature. This in turn implies that at 1400 °C, CMC with 50 percent

SCS-6 fiber should be tough and not fail catastrophically for short times and applied composite stresses less than 0.5 GPa.

Regarding SiC fiber creep resistance, figure 7 is a plot of the stress dependence of the creep strain measured after 20 hr at 1300 °C for three possible CMC constituents. Included are data points for the Avco CVD fibers (ref. 13), for the Nicalon fiber (ref. 16), and for reaction-bonded silicon nitride (RBSN) at two densities (ref. 17). The dashed lines are extrapolations of the data points based on a $n = 1$ stress exponent for the SiC fibers (ref. 13) and $n = 2$ for the RBSN (ref. 17). The upper point of each line approximates the stress and strain at fracture for a 20-hr rupture life at 1300 °C.

Three points of practical significance can be drawn from figure 7. First, the creep resistance of the CVD fibers is almost an order of magnitude better than the Nicalon fiber. Second, for a CMC consisting of an RBSN matrix reinforced by CVD fibers, the fiber fracture strain is greater than that of the matrix even under high temperature creep conditions. Thus, at least for this CMC system, the desirable toughening mechanism of fiber bridging of matrix macrocracks should operate both at low and high temperatures. Third, for the SiC/RBSN system, the fiber stiffness, that is, applied stress per total strain, remains greater than the matrix stiffness even at high temperatures. Thus, if processing and service-induced matrix flaws could be kept equivalent in size to typical processing and service-induced monolithic flaws, the CMC should be stronger and tougher than monolithic RBSN at all envisioned service temperatures.

At the present time, it would appear then in light of the above single fiber studies that a CVD-type SiC fiber similar in structure to the SCS-6 fiber offers the best opportunity for strong and tough CMC with potential application above 1200 °C. The possible loss in CMC structural performance due to the large diameter of the Avco fibers has yet to be determined. However, if the diameter drawback is found to be significant, the CVD production method offers the opportunity for fabricating fibers with measurably smaller diameters. In addition, the possibility exists for depositing thin coatings of various types on the base CVD fiber. This could be useful not only for optimizing high temperature environmental resistance but also for tailoring fiber-matrix interfacial bonding.

CONCLUDING REMARKS

Although at the present time very few, if any, commercial fibers meet all the property requirements for providing either MMC or CMC with high structural reliability, continued fiber research and development efforts should be successful in minimizing the property deficiencies of certain promising fibers. Indeed, the recent boron fiber success at NASA is a good example of how the strength deficiencies of large diameter boron fibers can be significantly reduced by fundamental research, thereby increasing the opportunities for tougher and more impact resistant MMC. For structurally reliable high temperature CMC, an area still in its infancy, although the polymer-derived SiC fiber lacks high stiffness and strength retention and the CVD SiC fiber lacks small diameter, technical approaches which can overcome these problems appear to be possible. It is in an attempt to develop these approaches that SiC fiber and

composite research efforts are currently in progress at NASA and other government agencies.

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TABLE I. - STRENGTH PROPERTIES OF LARGE DIAMETER BORON FIBERS
BEFORE AND AFTER CHEMICAL POLISHING

Diameter, μm (mils)	As-produced		Slight polish	
	Average strength, ^a GPa (ksi)	COV, ^b percent	Average strength, ^a GPa (ksi)	COV, ^b percent
203 (8)	4.0 (580)	7	4.4 (640)	3
280 (11)	3.6 (520)	12	4.2 (610)	4
406 (16)	2.1 (300)	14	4.6 (660)	4
Fracture sources	Surface, core		Core only	

^aGauge length = 25 mm.

^bCoefficient of variation = standard deviation/average value.

**TABLE II. - STRENGTH PROPERTIES OF IMPROVED LARGE
DIAMETER BORON FIBERS**

Diameter, μm (mil)	Treatment	Strength		Relative fracture energy
		Average ^a GPa (ksi)	COV, ^b percent	
142 (5.6)	As-produced	3.8 (550)	10	1.0
406 (16)	As-produced	2.1 (300)	14	0.3
382 (15)	Chemical polish	4.6 (660)	4	1.4
382 (15)	Oxygen plus polish	5.7 (820)	4	2.2

^aGauge length = 25 mm.

^bCoefficient of variation = standard deviation/average value.

TABLE III. - NICALON SiC FIBER - TYPICAL PROPERTIES

Production method:	Si and C-containing polymer spun, cured, and pyrolyzed
Diameter:	10 to 20 μm (500 per yarn)
Modulus:	180 GPa (420 GPa for β -SiC)
Strength at 20 °C:	
As-produced:	2 GPa
After 1400 °C:	<1 GPa
(Argon)	
Strength at 1400 °C:	<0.5 GPa
(oxygen)	
Creep strain	
at 1300 °C, 0.6 GPa, 20 hr:	4.5 percent

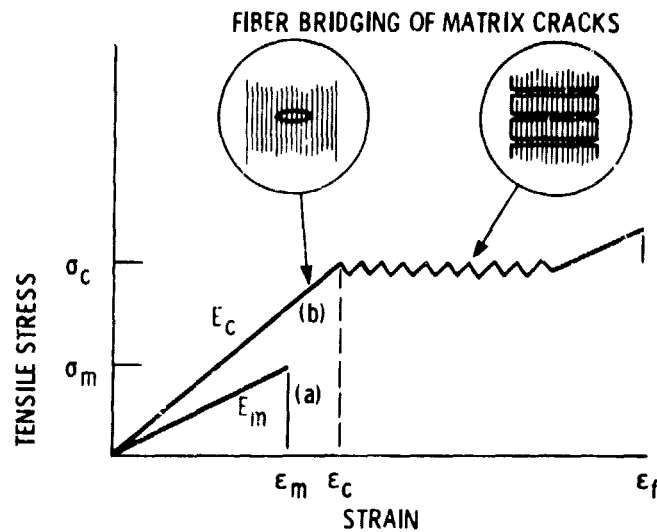


Figure 1. - Schematic of typical stress-strain curves for (a) monolithic ceramic and (b) CMC with continuous fibers aligned the load direction.

GENERAL:

- * CONTINUOUS (HIGH LENGTH/DIAMETER RATIO)
- * HIGH VOLUME FRACTION ALIGNED IN LOAD DIRECTION
- * HIGH AS-PRODUCED TENSILE STRENGTH
- * HIGH STRENGTH RETENTION UNDER COMPOSITE FABRICATION CONDITIONS
- * THERMAL EXPANSION NEAR THAT OF THE MATRIX

SPECIFIC:

MMC (DUCTILE MATRIX):

STRONG MATRIX BONDING

- * LARGE DIAMETER

CMC (BRITTLE MATRIX):

- * HIGH MODULUS
- * CREEP RESISTANT
- * SMALL DIAMETER
- WEAK MATRIX BONDING
- * HIGH STRENGTH RETENTION IN OXYGEN AND OTHER SERVICE ENVIRONMENTS

Figure 2. - Fiber property requirements for structurally reliable MMC and CMC. Asterisks indicate those which are generally independent of matrix chemical and physical properties.

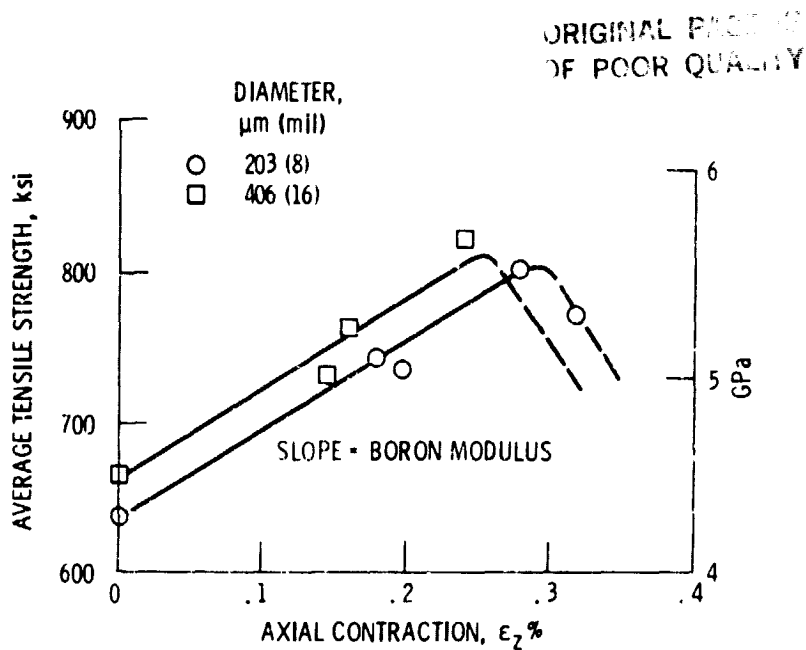


Figure 3. - The effects of oxidation-induced contraction (plus surface polishing) on the strength of large diameter boron fibers.

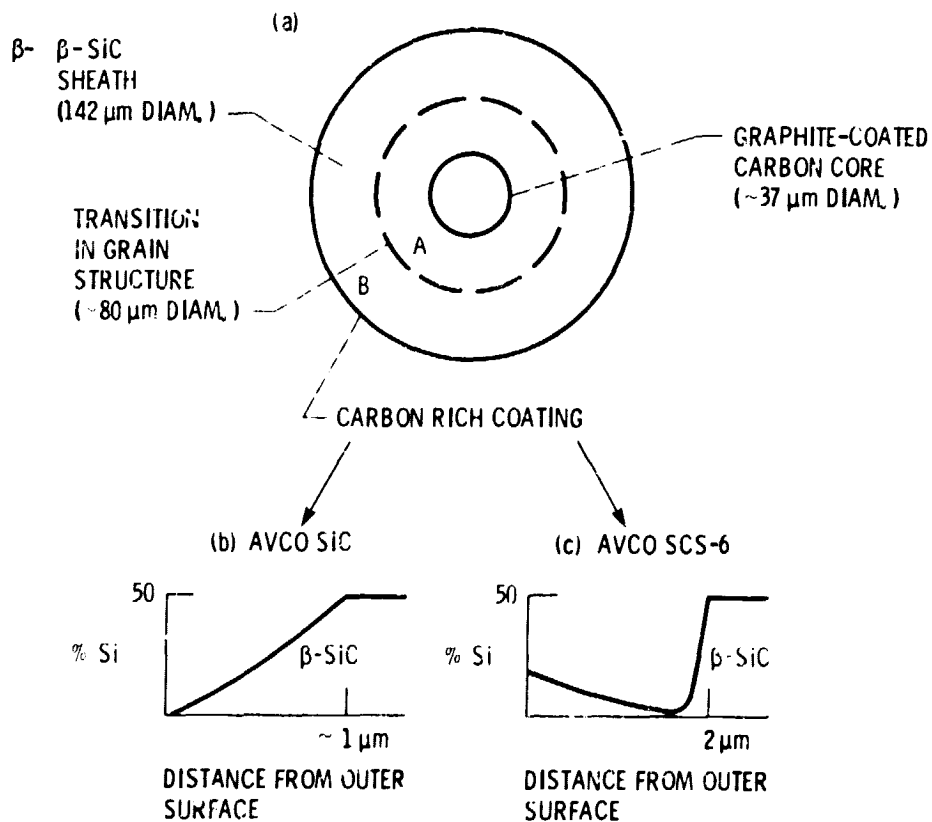


Figure 4. - Schematic representations of (a) the CVD SiC fiber cross section and (b and c) the silicon content in two types of fiber coatings (ref. 14).

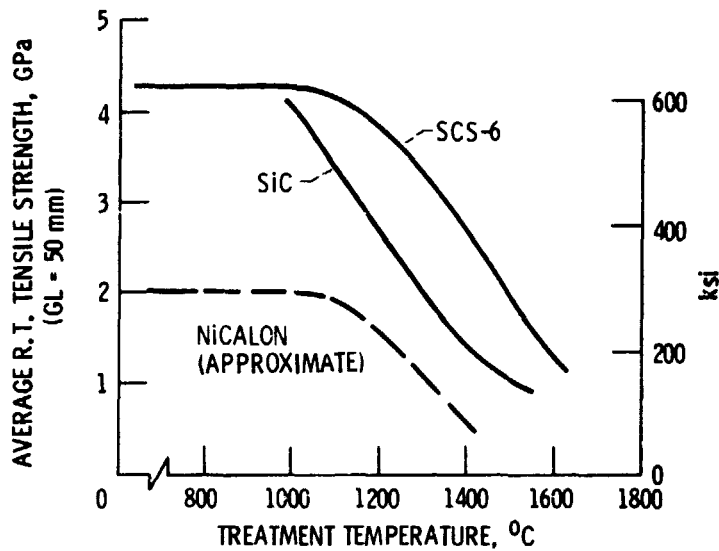


Figure 5. - Strength retention for SiC fibers in argon or nitrogen environments (15 min exposure).

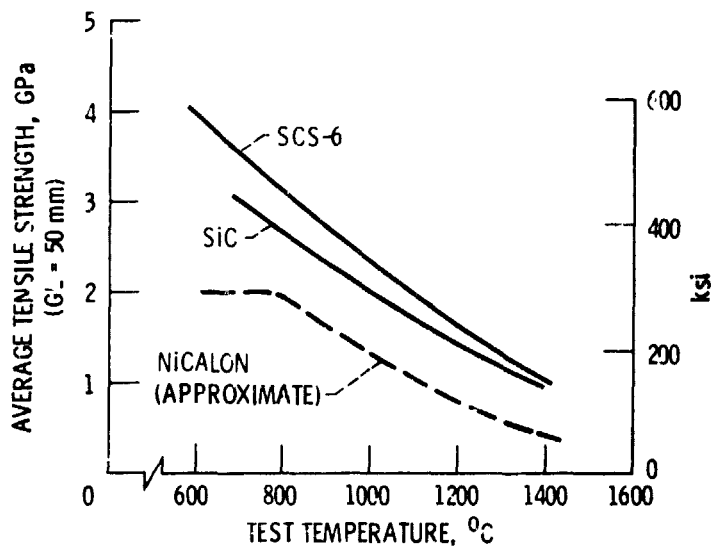


Figure 6. - Strength at temperature in oxygen for SiC fibers (15 min exposure).

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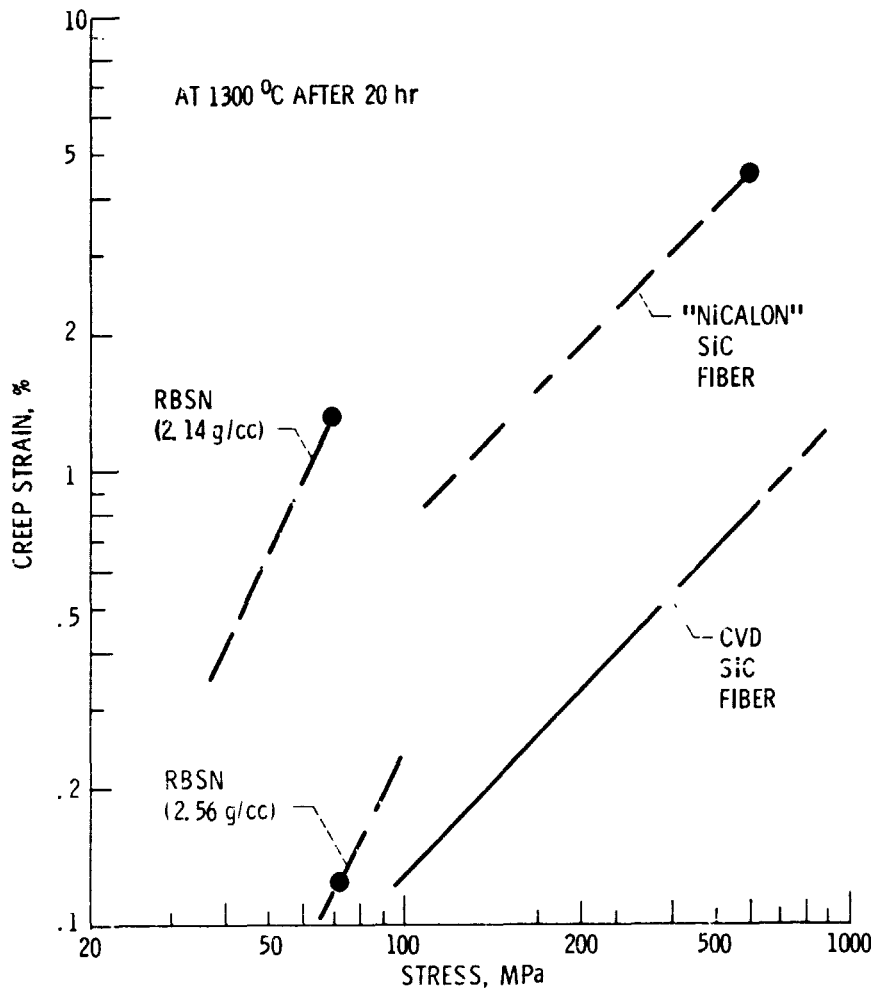


Figure 7. - A comparison of the stress-dependent creep of fiber and matrix materials with potential as constituents in high temperature ceramic matrix composites. Data points for RBSN, Nicalon, and CVD fiber are from references 17, 16, and 13 respectively.